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Term: electrospinning and apparatus

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L1: Entry 2 of 7 File: USPT Feb 18, 2003

DOCUMENT-IDENTIFIER: US 6520425 B1
TITLE: Process and apparatus for the production of nanofibers

Brief Summary Text (4):
It is known to produce nanofibers by using electrospinning techniques. These techniques, however, have been problematic because some spinnable fluids are very viscous and require higher forces than electric fields can supply before sparking occurs, i.e., there is a dielectric breakdown in the air. Likewise, these techniques have been problematic where higher temperatures are required because high temperatures increase the conductivity of structural parts and complicate the control of high electrical fields.

Brief Summary Text (5):
It is known to use pressurized gas to create polymer fibers by using melt-blowing techniques. According to these techniques, a stream of molten polymer is extruded into a jet of gas. These polymer fibers, however, are rather large in that the fibers are greater than 1,000 nanometers (1 micron) in diameter and more typically greater than 10,000 nanometers (10 microns) in diameter. It is also known to combine electrospinning techniques with melt-blowing techniques. But, the combination of an electric field has not proved to be successful in producing nanofibers inasmuch as an electric field does not produce stretching forces large enough to draw the fibers because the electric fields are limited by the dielectric breakdown strength of air.

Brief Summary Text (7):
Many nozzles and similar apparatus that are used in conjunction with pressurized gas are also known in the art. For example, the art for producing small liquid droplets includes numerous spraying apparatus including those that are used for air brushes or pesticide sprayers. But, there are no apparatus or nozzles capable of simultaneously producing a plurality of nanofibers from a single nozzle.

Drawing Description Text (2):
FIG. 1 is a schematic diagram of an apparatus for producing nanofibers according to this invention.

Drawing Description Text (3):
FIG. 2 is a schematic representation of a preferred embodiment of the apparatus of this invention, wherein the apparatus includes a lip cleaner assembly.

Drawing Description Text (4):
FIG. 3 is a schematic representation of a preferred embodiment of the apparatus of this invention, wherein the apparatus includes an outer gas shroud assembly.

Drawing Description Text (5):
FIG. 4 is a schematic representation of a preferred embodiment of the apparatus of the invention, wherein the apparatus includes an outer gas shroud, and the shroud is modified with a partition.

Drawing Description Text (7):
FIG. 6 is a schematic representation of a preferred embodiment of the apparatus of this invention wherein the apparatus is designed for batch processes.

Drawing Description Text (8):
FIG. 7 is a schematic representation of a preferred embodiment of the apparatus of this invention wherein the apparatus is designed for continuous processes.

Drawing Description Text (9):
FIG. 8 is a schematic representation of a preferred embodiment of the apparatus of this invention wherein the apparatus is designed for the production of a mixture of nanofibers from one or more polymers simultaneously.

Drawing Description Text (10):
FIG. 9 is a schematic representation of a preferred embodiment of the apparatus of this invention, wherein the apparatus includes an outer gas shroud assembly.

Drawing Description Text (11):
FIG. 10 is a schematic representation of another embodiment of the apparatus of the invention, wherein the apparatus includes an outer gas shroud, having a partition directed radially inward at an end thereof.

Detailed Description Text (4):
A nozzle 10 that is employed in practicing the process of this invention is best described with reference to FIG. 1. Nozzle 10 includes a center tube 11 having an entrance orifice 26 and an outlet orifice 15. The diameter of center tube 11 can vary based upon the need for gas flow, which impacts the velocity of the gas as it moves a film of liquid across the jet space 14, as will be described below. In one embodiment, the diameter of tube 11 is from about 0.5 to about 10 mm, and more preferably from about 1 to about 2 mm. Likewise, the length of tube 11 can vary depending upon construction conveniences, heat flow considerations, and shear flow in the fluid. In one embodiment, the length of tube 11 will be from about 1 to about 20 cm, and more preferably from about 2 to about 5 cm. Positioned concentrically around and apart from the center tube 11 is a supply tube 12, which has an entrance orifice 27 and an outlet orifice 16. Center tube 11 and supply tube 12 create an annular space or column 13. This annular space or column 13 has a width, which is the difference between the inner and outer diameter of the annulus, that can vary based upon the viscosity of the fluid and the maintenance of a suitable thickness of fiber-forming material fluid on the inside wall of gas jet space 14. In a preferred embodiment, the width is from about 0.05 to about 5 mm, and more preferably from about 0.1 to about 1 mm. Center tube 11 is vertically positioned within supply tube 12 so that a gas jet space 14 is created between lower end 24 of center tube 11 and lower end 23 of supply tube 12. The position of center tube 11 is adjustable relative to lower end 23 of supply tube 12 so that the length of gas jet space 14 is adjustable. Gas jet space 14, i.e., the distance between lower end 23 and lower end 24, is adjustable so as to achieve a controlled flow of fluid along the inside of tube 12, and optimal conditions for nanofiber production at the end 23 of tube 12. In one embodiment, this distance is from about 0.1 to about 10 mm, and more preferably from about 1 to about 2 mm. It should be understood that gravity will not impact the operation of the apparatus of this invention, but for purposes of explaining the present invention, reference will be made to the apparatus as it is vertically positioned as shown in the figures.

Detailed Description Text (6):
According to the present invention, nanofibers are produced by using the apparatus of FIG. 1 by the following method. Fiber-forming material is provided by a source 17, and fed through annular space 13. The fiber-forming material is directed into gas jet space 14. Simultaneously, pressurized gas is forced from a gas source 18 through the center tube 11 and into the gas jet space 14

Detailed Description Text (15):
According to this embodiment, nanofibers are produced by using the apparatus of FIG. 6 according to the following method. Pressure is applied to the container so that fiber-forming material is forced from storage space 35 into gas jet space 14. The pressure that is applied can result from gas pressure, pressurized fluid, or molten polymer from an extruder. Simultaneously, pressurized gas is forced from a gas source 18, through center tube 11, and exits through center tube orifice 15 into gas jet space 14. As with previous embodiments, heat may be applied to the fiber-forming material prior to or after being placed in fiber-forming material container 34, to

the pressurized gas entering center tube 11, and/or to storage space 35 by heat source 39 or additional heat sources. Fiber-forming material exiting from storage space 35 into gas jet space 14 forms a thin layer of fiber-forming material on the inside wall of gas jet space 14. This layer of fiber-forming material is subjected to shearing deformation, or other modes of deformation such as surface wave, by the gas jet until it reaches container outlet orifice 36. There the layer of fiber-forming material is blown apart, into many small strands, by the expanding gas.

Detailed Description Text (16):
In still another embodiment, as shown in FIG. 7, the fiber-forming material can be delivered on a continuous basis rather than a batch basis as in FIG. 6. In this embodiment, the apparatus is a continuous flow nozzle 41. Consistent with previous embodiments, nozzle 41 comprises a center tube 11, a supply tube 12, an outer gas tube 19, and a gas shroud tube 31. Supply tube 12 is positioned concentrically around center tube 11. Outer gas tube 19 is positioned concentrically around supply tube 12. Gas shroud tube 31 is positioned concentrically around outer gas tube 19. Center tube 11 has an entrance orifice 26 and an outlet orifice 15. As in previous embodiments, the diameter of center tube 11 can vary. In one embodiment, the diameter of tube 11 is from about 1 to about 20 mm, and more preferably from about 2 to about 5 mm. Likewise the length of tube 11 can vary. In a preferred embodiment, the length of tube 11 will be from about 1 to about 10 cm, and more preferably from about 2 to about 3 cm.

Detailed Description Text (21):
According to the present invention, nanofibers are produced by using the apparatus of FIG. 7 by the following method. Fiber-forming material is provided by a source 17 through supply inlet tube 51 into and through annular space 13, and then into gas jet space 14. Preferably the fiber-forming material is supplied to the supply inlet tube 51 under a pressure of from about 0 to about 15,000 psi, and more preferably from about 100 to about 1,000 psi. Simultaneously, pressurized gas is forced through inlet tube 52, through center tube 11, and into gas jet space 14. As with previously described embodiments, it is believed that fiber-forming material is in the form of an annular film within gas jet space 14. This layer of fiber-forming material is subjected to shearing deformation by the gas jet exiting from the center tube outlet orifice 15 until it reaches the fiber-forming material supply tube outlet orifice 16. At this point, it is believed that the layer of fiber-forming material is blown apart into many small strands by the expanding gas. Once ejected from orifice 16, these strands solidify in the form of nanofibers. This solidification can occur by cooling, chemical reaction, coalescence, ionizing radiation or removal of solvent. As with previously described embodiments also simultaneously, pressurized gas is supplied by gas source 25 to lip cleaner inlet tube 53 into outer gas tube 19.

Detailed Description Text (30):
Nanofibers are produced by using the apparatus of FIG. 8 by the following method. A first fiber-forming material is provided by a first material source 94, and fed through first annular space 69 and directed into first gas jet space 71. Pressurized gas is forced from a gas source through the center tube 11 and into first gas jet space 71. This gas should be forced through center tube 11 at a sufficiently high pressure so as to carry the fiber forming material along the wall of jet space 71 and create nanofibers, as mentioned in previous embodiments. A second fiber-forming material may be provided by the first material source (not shown) or by a second material source 96, and fed through second supply annular space 79. The second fiber-forming material is directed into second gas jet space 92. Pressurized gas is forced from a source through middle gas annular column 75 and into second gas jet space 92. This gas should be forced through middle gas annular column 75 at a sufficiently high pressure so as to carry the fiber forming material along the wall of jet space 92 and create nanofibers, as mentioned in previous embodiments. Therefore, in one embodiment, the gas is forced through center tube 11 and middle gas tube 73 under a pressure of from about 10 to about 5,000 psi, and more preferably from about 50 to about 500 psi.

Detailed Description Text (35):
It should be understood that there are many conditions and parameters that will impact the formation of fibers according to the present invention. For example, the

pressure of the gas moving through any of the columns of the apparatus of this invention may need to be manipulated based on the fiber-forming material that is employed. Also, the fiber-forming material being used or the desired characteristics of the resulting nanofiber may require that the fiber-forming material itself or the various gas streams be heated. For example, the length of the nanofibers can be adjusted by varying the temperature of the shroud air. Where the shroud air is cooler, thereby causing the strands of fiber-forming material to quickly freeze or solidify, longer nanofibers can be produced. On the other hand, where the shroud air is hotter, and thereby inhibits solidification of the strands of fiber-forming material, the resulting nanofibers will be shorter in length. It should also be appreciated that the temperature of the pressurized gas flowing through center tube 11 and middle gas tube 73 can likewise be manipulated to achieve or assist in these results. For example, acicular nanofibers of mesophase pitch can be produced where the shroud air is maintained at about 350.degree. C. This temperature should be carefully controlled so that it is hot enough to cause the strands of mesophase pitch to be soft enough and thereby stretch and neck into short segments, but not too hot to cause the strands to collapse into droplets. Preferred acicular nanofibers have lengths in the range of about 1,000 to about 2,000 nanometers.

Detailed Description Text (37):
In one specific embodiment the present invention, carbon nanofiber precursors are produced. Specifically, nanofibers of polymer, such as polyacrylonitrile, are spun and collected by using the process and apparatus of this invention. These polyacrylonitrile fibers are heated in air to a temperature of about 200 to about 400.degree. C. under tension to stabilize them for treatment at higher temperature. These stabilized fibers are then converted to carbon fibers by heating to approximately 1700.degree. C. under inert gas. In this carbonization process, all chemical groups, such as HCN, NH.sub.3, CO.sub.2, N.sub.2 and hydrocarbons, are removed. After carbonization, the fibers are heated to temperatures in the range of about 2000.degree. C. to about 3000.degree. C. under tension. This process, called graphitization, makes carbon fibers with aligned graphite crystallites.

Detailed Description Text (39):
In another embodiment, NGJ is combined with electrospinning techniques. In these combined process, NGJ improves the production rate while the electric field maintains the optimal tension in the jet to produce orientation and avoid the appearance of beads on the fibers. The electric field also provides a way to direct the nanofibers along a desired trajectory through processing machinery, heating ovens, or to a particular position on a collector. Electrical charge on the fiber can also produce looped and coiled nanofibers that can increase the bulk of the non-woven fabric made from these nanofibers.

Detailed Description Text (42):
It should also be appreciated that the average diameter and the range of diameters is affected by adjusting the gas temperature, the flow rate of the gas stream, the temperature of the fluid, and the flow rate of fluid. The flow of the fluid can be controlled by a valve arrangement, by an extruder, or by separate control of the pressure in the container and in the center tube, depending on the particular apparatus used.

Detailed Description Text (43):
It should thus be evident that the NGJ methods and apparatus disclosed herein are capable of providing nanofibers by creating a thin layer of fiber-forming material on the inside of an outlet tube, and this layer is subjected to shearing deformation until it reaches the outlet orifice of the tube. There, the layer of fiber-forming material is blown apart, into many small jets, by the expanding gas. No apparatus has ever been used to make nanofibers by using pressurized gas. Further, the NGJ process creates fibers from spinnable fluids, such as mesophase pitch, that can be converted into high strength, high modulus, high thermal conductivity graphite fibers. It can also produce nanofibers from a solution or melt. It may also lead to an improved nozzle for production of small droplets of liquids. It should also be evident that NGJ produces nanofibers at a high production rate. NGJ can be used alone or in combination with either or both melt blowing or electrospinning to produce useful mixtures of fiber geometries, diameters and lengths. Also, NGJ can be used in conjunction with an electric field, but it should be appreciated that an

electric field is not required.

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1. Document ID: US 6520425 B1

L3 Entry 1 of 2 File: USPT Feb 18, 2003

US-PAT-NO: 6520425
DOCUMENT-IDENTIFIER US 6520425 B1

TITLE: Process and apparatus for the production of nanofibers

DATE-ISSUED: February 18, 2003

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
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US-CL-CURRENT: 239/294; 239/423, 239/424

Full | Title | Citation | Front | Review | Classification | Date | Reference | Sequences | Attachments | Claims | KWIC | Draw Desc | Image

2. Document ID: US 6382526 B1

L3: Entry 2 of 2 File: USPT May 7, 2002

US-PAT-NO: 6382526
DOCUMENT-IDENTIFIER: US 6382526 B1

TITLE: Process and apparatus for the production of nanofibers

DATE-ISSUED: May 7, 2002

INVENTOR-INFORMATION:

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US-CL-CURRENT: 239/294; 239/424

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